

Plant Synthesis: Garlic

Used to synthesize gold nanoparticles ranging from 1-10nm.

Protocol:

Garlic aqueous plant extraction

1. Peel garlic cloves and wash thoroughly with tap water.
2. Rinse garlic cloves with ionized water, then dry them with a paper towel.
3. Weigh out approximately 3 g of garlic cloves. Cut the cloves with scissors or a knife into smaller pieces in order to more accurately weight to 3 g.
4. Grind the weighed 3 g of garlic cloves using a mortar and pestle.
5. Filter the ground garlic using a Whatman filter 1 paper and glass funnel into a 100 mL Erlenmeyer flask with 30 mL of MilliQ water. Set up a vacuum apparatus to aid transfer of water-garlic extract into the flask.
6. The garlic extract solution collected in the flask is then passed through a 0.45 micron Whatman filter 1 paper-covered funnel into a new clean flask to further filter the liquid extract. This is repeated twice.
7. Store the garlic extract solution in glass (Duran) bottles at 4°C in fridge.
 - Do NOT place the garlic extract solution in a freezer (at -20°C or -80°C) - freezing the solution will alter the active ingredient in the garlic extract and decrease yield of nanoparticles during synthesis

Synthesis of gold nanoparticles using aqueous garlic extract solution

1. Preset water double boiler to 95°C. Set up the double boiler in a fume hood using a hot plate and a large beaker filled with tap water. Use a mercury thermometer to monitor the temperature of the water bath.
2. Make 20 mL of a 1% garlic extract solution.
 - Using a graduated cylinder, measure 19.8 mL of ddH₂O and pour into a 100 mL beaker
 - Micropipette 200 uL (0.2 mL) of garlic extract solution into water in the beaker
 - Place the beaker on a stir plate and add a magnetic stir bar to the beaker to allow the mixture to stir and become homogeneous
3. Prepare 67 uL of 75 mM HAuCl₄ solution.
 - A. Weigh out 2.55 mg (0.00255g) of chloroauric acid trihydrate (HAuCl₄) solid on a weigh-boat.
 - CAUTION: oxidizes quickly - do not expose to air for longer than required
 - Do NOT use metal scoopula - use plastic or wood to transfer the solid
 - LIGHT SENSITIVE - use for short duration under light conditions
 - Note: chloroauric acid is pricey - try not to be wasteful!
 - B. Using a micropipet, quickly add ~100 uL of the ddH₂O from the flask to the crystals of chloroauric acid trihydrate on the weigh-boat. This should dissolve the solid to generate a yellow mixture on the weigh-boat.
4. Use the micropipette to transfer the 67 uL of chloroauric acid solution to the 20 mL garlic solution in the beaker. Stirring should be continuous during this process to ensure dispersion of the HAuCl₄.
5. Use pH paper to determine the pH of the garlic-HAuCl₄ solution.
6. Add NaOH (40%) very slowly dropwise to the mixture in the beaker until a pH ~10 with stirring. Wait several seconds between the addition of each drop of NaOH. This gives time for the NaOH to equilibrate in the mixture and stabilize the overall pH of the solution.
 - A basic pH is required to allow for the nanoparticle synthesis reaction to occur.

7. Fix the beaker containing the mixture in the makeshift water bath by immersing the lower half of the beaker in the heated water.
 - Use the clamp of a ring stand in the fume hood to stabilize the setup.
 - Nanoparticle synthesis occurs at this step - a color change of solution to pink or purple indicates nanoparticle formation
8. Allow up to 20 minutes for nanoparticle synthesis reaction
 - At approximately 7-12 mins, if a significant color change is seen, remove the beaker from the water bath
 - Keeping the beaker for longer in the water bath past color change may cause nanoparticle aggregation and generation of oversized gold particles
9. In a small sealed vial wrapped in aluminum foil, the nanoparticle solution can be stored in a 4°C fridge.
 - Covering the vial in aluminum foil prevents the nanoparticles from being exposed to light which will prolong the shelf-life and integrity of the particles

Source: Rastogi, L., & Arunachalam, J. (2013). Green synthesis route for the size controlled synthesis of biocompatible gold nanoparticles using aqueous extract of garlic (*allium sativum*). *Advanced Materials Letters*.